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DETROIT

The Metallurgist.

Supplement to The Engineer, *December, 1937.*

Pure Cementite.

INVESTIGATORS who have attempted to isolate cementite by simple acid attack or anodic dissolution of ferrous materials have rarely obtained a residue having a carbon content corresponding to Fe_3C (*i.e.*, 6·67 per cent.), but usually one of higher carbon content. The tendency to high carbon content was noted by Arnold and Read in 1894. They attributed it to the decomposition of carbide, accompanied by liberation of carbon which proceeds simultaneously with the anodic dissolution. Ruff and Gersten suggested the separation of carbon by flotation in heavy liquids. This method has been applied by A. Travers and R. Diebold, who have described their results in a note[1] "On the Isolation of Pure Cementite by Acid Attack of Ferrous Materials, and on Some Physical Properties of this Substance." The dense residue obtained in the attack of a very pure white iron, containing 3·4 per cent. of carbon with only traces of manganese and 0·3 per cent. of silicon, was washed several times with alcohol, and then treated with freshly distilled trichlorethylene (density 1·47). The heavy residue was finally washed successively with alcohol and ether, and was dried in nitrogen. The product obtained was friable, fine grained, and unctuous to the touch. The carbide isolated by the attack of normal acetic acid solution was steel grey, that prepared by anodic attack (Arnold and Read's method) showed a slight pale yellow iridescence, suggesting that possibly a slight oxidation is produced during anodic attack. Analysis of the residue was carried out under low oxygen pressure by Ströhlein's method, the volume of CO_2 produced being measured. The carbon content was found to be 6·6 per cent., with a relative error of about 1 per cent. (*i.e.*, $\pm 0·07$).

Several properties of this pure carbide were investigated. The magnetic change point (Curie point), to which various temperatures from 180 deg. to 215 deg. Cent. have been assigned, was found to occur at 210 deg. Cent., a temperature in exact agreement with the anomaly in dilatation noted by Chevenard and the discontinuity in calorimetric measurements observed by Wever. Electrolytic potential measurements were made under an atmosphere of nitrogen from which oxygen was excluded, a massive electrode produced from the carbide powder in a hydraulic press being used. The electrode potential was stated to be about $-0·32$ volt for pH=2·3, and $-0·50$ for pH=7, returning towards the first-mentioned value as the pH increased to 7·5. The cementite was markedly positive to iron and the couple Fe_3C-Fe attained an E.M.F. of 0·23 volt in a neutral medium at 18 deg. Cent.

Some criticism is levelled by the authors against Westgren and Phragmén, who were the first to obtain an X-ray spectrum of cementite, on the grounds that their material contained excess carbon. The authors claim that their own pure product gives a sharp X-ray spectrum, which is reproduced. In itself, however, it is of little value as no quantitative data are provided. Moreover, the criticism cannot be regarded as effective in view of the recent demonstration by H. Gröber and H. Hanemann[2] that cementite has no appreciable solubility for iron or for carbon, and that a slight admixture of either would therefore not introduce lattice distortion. Gröber and Hanemann have described experiments in which they prepared from Armco iron and sugar charcoal, superheated and rapidly chilled, uniform non-graphitic iron-carbon alloys of high purity, containing up to 6·67 per cent. of carbon, and alloys with still more carbon, up to 7·7 per cent., containing cementite and graphite. The cementite X-ray spectra obtained from these alloys showed deviations which lay within the limits of possible error. The lattice constants of pure cementite at room temperature, deduced from these spectra, were

$$a = 4·51 \pm 0·005 \text{ Å}.$$
$$b = 5·06 \pm 0·005.$$
$$c = 6·72 \pm 0·01.$$

The results obtained on material with excess carbon by Westgren and Phragmén (1924), viz., 4·518, 5·069, and 6·736 Å, show good agreement with these values.

[1] *Comptes Rendus*, November 3rd, 1937, 205, 797; also "The Decomposition of Pure Cementite by Acids," *ibid*, November 15th, 1937, 205, 916.

Titanium Alloys.

THERE is a large and increasing field of application of titanium as a minor constituent of a number of alloys both ferrous and non-ferrous. Typical uses, of this kind have been summarised by Comstock,[1] and later by Duma, but alloys in which titanium is the major constituent are rare.

Titanium is widely distributed, though generally in rather small quantities, and if a demand arose the metal, which is actually about the tenth commonest element in the earth's crust, need not remain a rare product. As usually obtained, metallic titanium is brittle owing to the presence of impurities. On the other hand, a metal which, when produced from purified materials,[2] is malleable when hot and can to some extent be bent at room temperatures can be made by the reduction of titanium tetrachloride with sodium

[2] *Archiv für das Eisenhüttenwesen*, October, 1937, 1937-38, 11, 199.

in a steel bomb[3] or by the dissociation of the iodide on a heated filament.[4] Although the sodium reduction method is stated to be capable of giving titanium of 98–99 per cent. purity on a semi-commercial scale,[5] neither of these processes is suitable for the production of large amounts of titanium.

A titanium of high purity and satisfactory hot-working qualities has been prepared by W. Kroll by reduction of the oxide, and the use of this material to study the properties of some titanium-rich alloys has been described.[6] The method of production of the titanium in a workable condition consists of mixing pure titanium oxide with shavings of metallic calcium and a powdered fused 75 : 25 mixture of calcium and barium chlorides. This is pressed into briquettes and heated electrically at 1000 deg. Cent. in a specially designed argon-filled furnace; 770 g. TiO_2 + 770 g. Ca + 1000 g. $CaCl_2/BaCl_2$ (75 : 25) at 1000 deg. Cent. for three hours gave 348 g. Ti (yield 76 per cent. of the theoretical). This product still contained sufficient oxygen to make it brittle. It was further reduced by the additional action of calcium hydride: 348 g. Ti (from the previous experiment) + 400 g. $CaCl_2/BaCl_2$ (75 : 25) + 50 g. Ca + 50 g. CaH_2 at 1000 deg. Cent. for one hour yielded 337 g. Ti. The resultant product was fairly uniform in a strongly sintered melt which was treated with water and strong hydrochloric acid to disintegrate it into titanium powder. This powder, after sintering *in vacuo*, was deformable when hot, but was more or less brittle when cold. The alloying additions were as pure as possible in order that no detriment to the titanium should result from the introduction of a non-deformable metal. All the additions were themselves malleable except silicon (99·4 per cent.) and beryllium (99·5 per cent.). The titanium powder was mixed with from 2 to 9 per cent. of the added element, pressed at 2000 atmospheres, sintered and degassed *in vacuo* at high temperature, except when the added element was volatile under these conditions. The alloys were then heated in argon at a pressure of about 50 mm. up to nearly the melting point, high-frequency current being employed as in the same author's research on the deformable alloys of chromium.[7]

Sintered corundum was found to be the most satisfactory lining, since it only reacts with titanium at temperatures above the melting point of the metal. The oxides of beryllium, zirconium, and thorium are readily reduced by titanium; so also is magnesia and the magnesium formed volatilises violently. So far no non-reacting crucible lining for the melting of titanium is known, and only a sintering process is at present possible for manufacture of the alloys. Some information about fusibility was, however, obtained. The melting point of titanium is about 1800 deg. Cent. It is raised by the addition of 5 per cent. of molybdenum or of tungsten, and lowered to about 1500 deg. Cent. by a similar amount of iron, nickel, or cobalt. All the other additions act similarly in lowering the melting point, the most effective being beryllium, 2 per cent. of which reduces the melting point to 1300 deg. Cent.

The sintered alloys were reheated to 800 deg. Cent. in a salt bath containing KCl 40, NaCl 40, and $CaCl_2$ 20 per cent. and were rolled into sheets. The sintered alloys were flat cylinders 21 mm. diameter and 6·5 mm. thick. They were rolled to elliptical sheet 1·5 mm. thick. The workability of the alloys of titanium with nickel, iron, and cobalt was found to be excellent; the sheet from these alloys was smooth and free from defects. Good rolling properties were also shown by alloys with tantalum, molybdenum, tungsten, manganese, chromium, and vanadium. A rough and speckled surface was shown by the alloys containing either copper, aluminium, zirconium, or silicon, and the alloy containing beryllium was completely red short. Some light was thrown on this behaviour by an examination of the microstructure after etching with dilute hydrofluoric acid. Nickel, iron, and copper alloys (hardness 363 to 477) showed titanium crystals embedded in solid solution. The molybdenum, tungsten, and tantalum alloys were uniform solid solutions (hardness 363). The manganese, chromium, and vanadium alloys (hardness 410, 451, and 477) showed titanium-rich crystals with a grain boundary constituent. All the others showed the presence of a compound and a grain boundary constituent. The 2 per cent. beryllium alloy was the softest (229) and had the worst working properties, being much inferior to the 2 per cent. silicon alloy, the hardness of which was 477. There are no indications of the possibility of application of these alloys except the statement that the alloys with copper, silicon, and tantalum were particularly difficult to etch. Of these, the tantalum alloy is by far the most malleable.

The presence of oxygen was found to have a very deleterious influence on the hot-workability of titanium. Its occurrence in the form of oxide films and inclusions also had a marked embrittling effect on the cold metal.

To his work on workable chromium,[7] vanadium, thorium, and uranium[8] Kroll has now added this study of the deformable titanium alloys. Although practical application of these pure metals and alloys is still far distant, this exploratory work is an essential stage in the developments ultimately to be expected.

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[1] See THE METALLURGIST, June, 1935, 1935–36, 10, 47; also J. A. Duma, "Effect of Titanium on Some Cast Ferrous and Non-Ferrous Metals," *Trans. Amer. Soc. for Metals*, Sept., 1937, 25, 788.

[2] W. Kroll, *Metallwirtschaft*, 1930, 9, 1043; A. E. van Arkel, *Metallwirtschaft*, 1934, 13, 405, 511.

[3] M. A. Hunter, *Journal*, Amer. Chem. Soc., 1910, 32, 330.

[4] A. E. van Arkel and J. H. de Boer, *Zeit. anorg. Chem.*, 1925, 148, 345.

[5] M. A. Hunter and A. Jones, *Trans. Electrochem. Soc.*, 1934, 66, 21.

[6] "Deformable Titanium and Zirconium," *Zeit. anorg. Chem.*, 1937, 234, 42; "Deformable Alloys of Titanium," *Zeit. Metallkunde*, 1937, 29, 189.

[7] *Zeit. Metallkunde*, 1936, 28, 317.

[8] *Ibid.*, page 30.

Recovery of Duralumin from Tensile Overstrain.

By R. H. GREAVES, D.Sc.

THE tensile overstrain and recovery of aluminium has been studied by Morley and Tomlinson.[1] They found that the limit of proportionality was decreased to zero and the extension for a given load greatly increased by overstrain. On reloading after a period of resting, the load extension diagram was restored to its original slope, recovery to the original modulus being complete after about fourteen days at atmospheric temperature and after 3 min. at 100 deg. Cent. Progress to completion was noticeable after one hour at atmospheric temperature, and was far advanced in five days. These results apply to rolled alumi-

[1] *Phil. Mag.*, 1906 (Sixth Series), 11, 380.

nium stressed to 11 tons per square inch and retested to 5·5 tons per square inch.

The behaviour noted by Morley and Tomlinson is dependent on the maximum load applied in reloading, being much less than the overstraining load. In experiments carried out under the conditions described in a previous paper,[2] overstrained aluminium always gave some slight additional permanent set when reloaded up to the overstraining load, whether tested immediately after overstrain or after resting at atmospheric temperatures for periods up to two months. The same was true of the annealed or hot-

the Young's modulus over the full range being 4600 tons per square inch.

A series of experiments was made to determine the conditions of recovery of duralumin from overstrain. For these tests ¾in. diameter rod was used, having the composition :—

Copper	4·3 per cent.
Manganese	0·7 ,,
Magnesium	0·4 ,,
Iron...	0·5 ,,
Silicon	0·4 ,,
Aluminium (by difference)	93·7 ,,

The alloy in the form of 5in. lengths was heated for

TABLE.—*Recovery of Duralumin from Tensile Overstrain.*

No. of test.	Condition of test piece.	Limit of proportionality.*	Load giving 0·2 per cent. permanent extension.*	Maximum load.†	Elongation on 2in.†	Reduction of area.†
		Tons per square inch.			Per cent.	
	Effect of ageing without overstrain :					
1	Quenched—Tested immediately	4	7·0	18·6	25	42
2	Aged 4 days	8	12·7	24·3	22	34
3	Aged 4 months	10·5	14·7	24·9	22	31
	Effect of ageing after overstrain of quenched alloy :‡					
1	Quenched—Tested immediately	4	7·0	18·6	25	42
4	Overstrained 2 per cent.—Tested immediately	10	13·3	19·7	23	36
5	Tested after 4 months	12·5	16·6	22·9	20	37
6	5 per cent.—Tested immediately	10	16·7	20·0	18	34
7	Tested after 4 months	13	18·5	23·5	19	37
	Effect of ageing after overstraining the quenched and aged alloy :					
3	Quenched and aged 4 months	10·5	14·7	24·9	22	31
8	Overstrained 2 per cent.—Tested immediately	12	17·5	24·2	23	34
9	Aged 1 day	14	18	25·6	25	33
10	7 days	15	18	24·5	24	36
11	14 days	16	18	24·8	24	33
12	Overstrained 5 per cent.—Tested immediately	11	22	24·3	22	35
13	Aged 1 day	16	21·5	24·5	24	33
14	7 days	17	21	24·5	22	29
15	14 days	17	22·5	24·6	22	29
	Effect of L.T.T. after overstraining the quenched and aged alloy :					
3	Quenched and aged 4 months	10·5	14·7	24·9	22	31
16	Overstrained 5 per cent.—15 min., 50 deg. Cent.	11	22·5	24·6	22	31
17	1 hour, 50 deg. Cent.	15	22	24·5	24	34
18	2 hours, 50 deg. Cent.	16	22	24·9	22	28
19	3 hours, 50 deg. Cent.	19	22	24·6	24	35
20	5 min., 75 deg. Cent.	10·5	21·5	24·5	24	33
21	15 min., 75 deg. Cent.	15	22	24·9	23	35
22	30 min., 75 deg. Cent.	19	21·5	25·1	23	35
23	1 hour, 75 deg. Cent.	14	20·5	24·9	23	33
24	5 min., 100 deg. Cent.	15	21	24·2	24	29
25	15 min., 100 deg. Cent.	12	21·5	24·8	25	35
	Effect of ageing after overstraining the quenched and tempered alloy:					
26	Quenched from 450 deg. Cent., heated 1 h. at 175 deg. Cent.	6	11	22·6	25	38
27	Overstrained 5 per cent.—Tested immediately	8	20·5	23·0	25	31
28	Aged 1 day	14	20·5	22·9	26	33
29	7 days	15	19·5	23·4	24	32
30	14 days	18	20·5	23·6	26	34
	Effect of L.T.T. after overstraining the quenched and tempered alloy:					
26	Quenched from 450 deg. Cent., heated 1 h. at 175 deg. Cent.	6	11	22·6	25	38
31	Overstrained 2 per cent., 30 min., 75 deg. Cent.	14	14·5	22·8	24	34
32	Overstrained 5 per cent. 30 min., 75 deg. Cent.	17	19·2	22·4	25	37

* Calculated on the cross-sectional area of the test piece at the beginning of each tensile test.
† Calculated on the original dimensions of the test piece before overstrain (if any).
‡ The test pieces were kept in ice until required for overstraining (maximum time, 1½ hours).

rolled aluminium alloys examined, but quenched and aged duralumin behaved differently in that a few days' rest was sufficient to restore elasticity up to the overstraining load, though longer was required to obtain maximum recovery. In accordance with the very slight development of hysteresis looping in overstrained duralumin, an 8in. test specimen of duralumin, reloaded immediately to the load giving 1 per cent. overstrain, viz., 15·5 tons per square inch, was found to show an additional permanent set of 22×10^{-5} inch, and reloaded after resting one day a further additional permanent set of only 2×10^{-5} inch,

[2] THE METALLURGIST, Oct. 29th, 1937, p. 72.

one hour at 450 deg. Cent., and quenched in water from that temperature.[3] With the exception of a few specimens, which were to be tested immediately and were kept in ice until required, the quenched alloy was either fully aged before being tested or low-temperature treated at 175 deg. Cent. The tests were made on 2in. gauge lengths by means of the Ewing extensometer reading to 8×10^{-6} inch. Results are given in the table and are illustrated by a series of curves on page 84.

Maximum load, elongation, and reduction of area

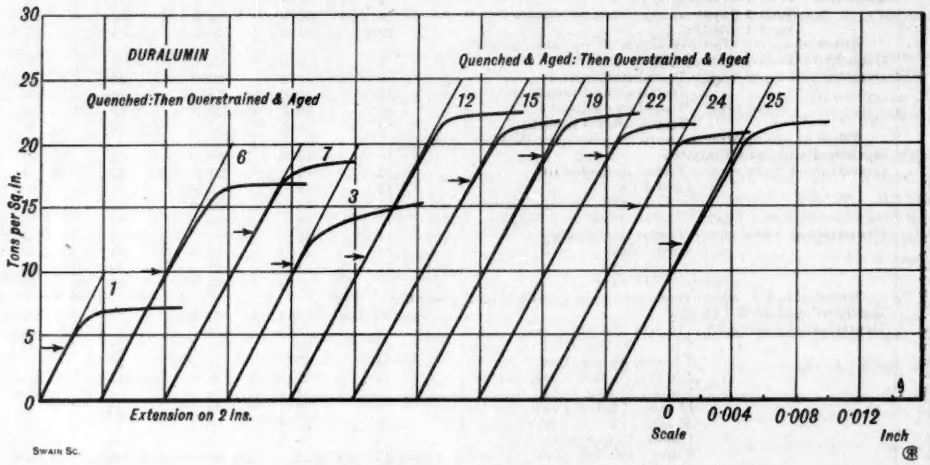
[3] To conform with modern practice a higher temperature should have been used.

were calculated on the original dimensions of the test piece. They show that, except when ageing of the quenched alloy is in progress, the final values so expressed are not affected by intervening overstrain and recovery treatments. The limit of proportionality and proof stress (load giving 0·2 per cent. permanent extension) were calculated on the cross-sectional area of the test piece at the beginning of each tensile test, *i.e.*, in the case of overstrained material on the cross-sectional area after overstrain. The immediate effect of overstrain was to raise the load giving 0·2 per cent. permanent extension, without lowering the limit of proportionality. Ageing or low-temperature treatment had little further effect on the 0·2 per cent. proof stress, but raised the limit of proportionality. After 2 per cent. overstrain the limit of proportionality had probably reached its maximum value in 14 days. After 5 per cent. overstrain, it was probably still rising at the end of 14 days, and a rather better figure was obtained after three hours at 50 deg. Cent., or 30 min. at 75 deg. Cent.; but higher temperatures failed to give as great an increase in limit of proportionality. A harder product was obtained by overstraining the quenched and aged alloy than by giving the same amount of overstrain to the quenched alloy, and subsequently ageing it, the load required to produce the same amount of overstrain being, of course, much lower in the case of the quenched alloy (*cf.* tests 5 and 11, 7 and 15). This is in accordance with the well known fact that plastic deformation subsequent to age-hardening results in mechanical properties which are superior to those obtained if the deformation precedes ageing.[4]

These tests were not specially designed for accurate determinations of Young's modulus, but it may be noted, in agreement with the results given in the previous paper, that the observed values fall between 4800 and 4550 tons (10·75 and $10·2 \times 10^6$ lb.) per square inch. The load extension curve for each test piece was obtained before overstraining, and the average value for the quenched and aged material was about 4700 tons ($10·5 \times 10^6$ lb.) per square inch. The low value (4550 tons, or $10·2 \times 10^6$ lb. per square inch) was confined to some of the specimens which had been overstrained 5 per cent.

[4] P. L. Teed, *Journ.*, Inst. Metals, 1936 (1), 58, 113.

Assistance was given, in carrying out these tests, by Mr. A. T. Cape, A.R.S.M.



Duralumin and Overstrain—Load Extension Diagrams. The numbers refer to the tests reported in the Table.

The Alloys of Iron and Zinc.

By W. D. JONES, M.Eng., Ph.E.

IN a recent review in THE METALLURGIST[1] upon the alloys of iron and zinc it was made clearly evident how much confusion there existed in the interpretation of reaction products of these two metals with respect to their binary equilibrium diagram, and how unsatisfactorily founded was the diagram which was available for the purpose. Since the publication of this review a number of important investigations have appeared, which have provided many new facts.[2 3 4]

It has for some time been considered as well established that the number and nature of reaction products or compound layers that form when a solid metal A is immersed in a molten metal B should agree with the number and nature of phases indicated by the binary equilibrium diagram of A and B. For some time there has been simultaneous doubt concerning the phases and layers in the iron-zinc diagram and in their reaction products, and in consequence no sound evidence was forthcoming to shake this seemingly well-established principle. In our last review, however, attention was drawn to evidence which for the first time, without regard to the nature of the alloys concerned, appeared to shake the foundations of this apparently well-established principle. This arose from the work of Scheil, Püngl, and Stenkhoff,[5 6] and of Musatti and La Falce,[7] all of whom showed that the rate of dissolution of iron in molten zinc, and the nature of the reaction products formed, could vary at constant temperature, a fact not explicable by the equilibrium diagram. The variations have for convenience been divided into two types, I and II, the rate of attack being much

greater in the case of type II and accompanied, in addition, by the disappearance of one or two phases or layers from the reaction products No explanation could be offered for this phenomenon at the date when this matter was last considered here, but it was suggested that impurities in the steel base might be an important consideration.

According to whether type I or II reactions are occurring, it is generally admitted that either three or two layers of reaction products are perceivable when iron is immersed in molten zinc, Figs. 2 and 4.

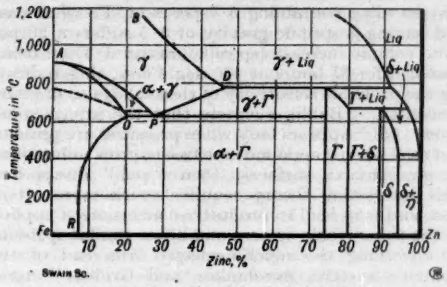


FIG. 1—*Iron-Zinc Diagram (Schramm).*

Since the most reliable equilibrium diagram available some months ago (that of Schramm, Fig. 1), only indicated two intermetallic phases Γ (between 20 and 27 per cent. Fe) and δ (between 6·3 and 11·5 per cent. Fe), then one of these layers had to be regarded as a mixture. Schramm, however, has recently provided a third phase ζ of narrow concentration limits at about 6 per cent. Fe (see Fig. 3), and has consequently greatly eased this difficulty. There can be little doubt of the authentic ty of the work of Schramm in this respect, since not only microscopic, but also

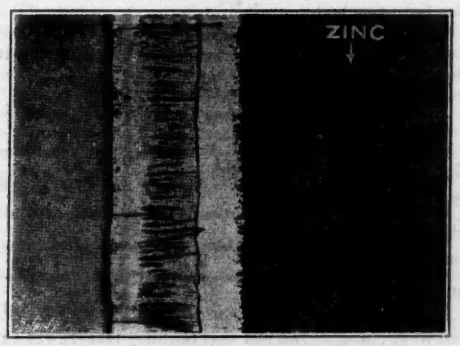


FIG. 2

careful X-ray, thermal, and magnetic investigations were embraced. It thus seems possible to name the three reaction layers that can be perceived in type I structures as Γ (nearest steel), δ (intermediate), and ζ (nearest zinc). Schramm has shown that the upper limit of stability of ζ is 530 deg. Cent., and it is reasonable to expect therefore the disappearance of this phase in structures obtained by reaction above this temperature. It is confusing, however, to find that it is the so-called δ that is most frequently found to disappear independently of temperature.

This division or identification of the three alloy layers into Γ, δ, and ζ has received the definite support of Scheil and Wurst,[8] who by the assistance of several methods and particularly by X-ray examination, claim to prove the presence of all three phases in type I structures in certain temperature ranges. These authors agree that ζ disappears at temperatures higher than 530 deg. Cent. and show that Γ

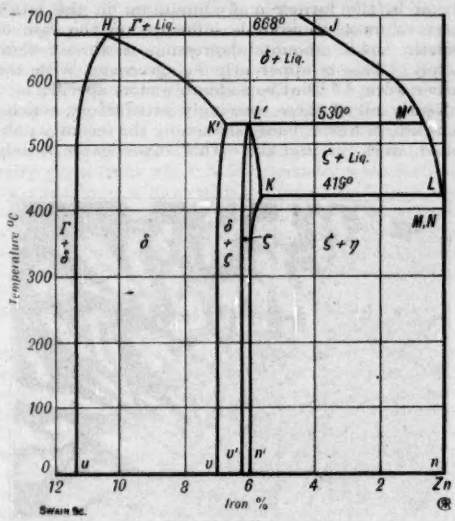


FIG. 3—*Iron-Zinc Diagram (Schramm).*

(next to the iron) is the mysteriously absent phase in type II structures. An explanation of this anomaly is attempted, based on the indisputable evidence that either type I or type II structures can be obtained dependent on whether the steel surface is flat or convex. In the case of a convex surface type II obtains, the rate of dissolution is higher, the layers

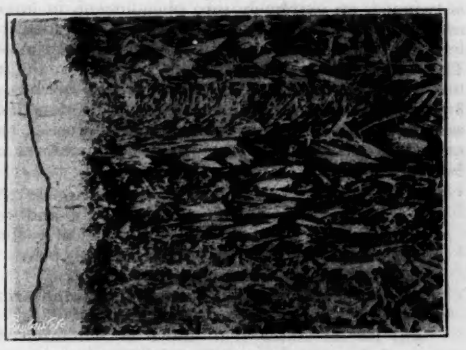


FIG. 4

thinner, and (according to these investigators), Γ is absent (see Fig. 5). It is assumed, and seems reasonable, that a convexity of growth permits the closer and more active approach of molten zinc to the steel base, aided possibly by capillary action in the middle layer, which is invariably cracked and porous. This theory is summarised by the statement that in type I growth of compound and dissolution of iron must

occur mainly by diffusion of iron through the compounds to the zinc, whilst in type II the principal transference must be that of zinc by capillary action towards the iron.

Scheil and Wurst have also established the facts that the rate and type of reaction are considerably influenced by the impurities in the steel base and in the molten zinc bath. The simultaneous presence of silicon in the former and aluminium in the latter exerts a most remarkable influence on the rate of growth, small amounts depressing it almost completely. This is apparently in agreement with the earlier work, [5 6 7] but considerably more specific.

Nearly all of these seemingly satisfactory conclusions are, however, badly shaken by the recently published work of Bablik. This investigator wisely

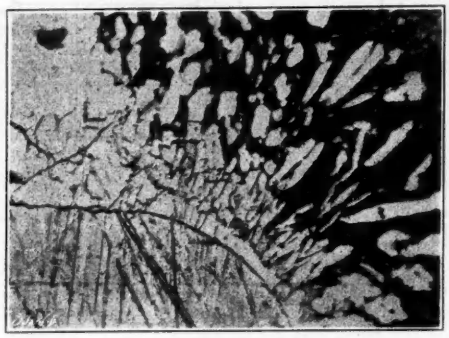


FIG. 5

refuses to offer definite designations for the three possible compound layers, but contents himself with the terms "an" (the layer nearest steel), "pal" (intermediate), and "ab" (nearest zinc).

Identification between the phases of Bablik's structures and those already considered is possible as follows:—The layer nearest the zinc, Bablik's "ab," is twinned, an extremely rare phenomenon in intermetallic compounds, and establishing the identity with Schramm's ζ phase, which is also shown twinned. The intermediate layer, Bablik's "pal," exhibits bad cracking, and this is the case also with the δ phase of Schramm. Here, however, the correspondence ends. In an endeavour to avoid confusion, the anomalies that now appear will be summarised as follows:—

(i) According to Bablik, if layers disappear the one that never disappears is "an" (Γ), and yet photographs in Bablik's paper show clearly that under these circumstances the remaining phase is twinned and should therefore be "ab" (ζ).

(ii) Scheil and Wurst claim that the disappearing phase is Γ.

(iii) Bablik states that under no condition do any of the alloy layers at any time contain more than 10·4 per cent. Fe, and if this is so then the presence of Γ cannot possibly be admitted in these alloys at all, which once again leaves us with one more layer than for which there are identified phases.

Thus once again these alloys are in a hopeless condition of confusion. Whilst such conditions exist, careful work having direct practical value, upon the rate of dissolution of iron in zinc, of the type recently published by Grubitsch and Brückner,[8 9] had better be postponed.

Bablik has an interesting and convincing explanation for the disappearing phase ("pal" or δ). He points out that Rawdon, Crossmann, and Finn[10] have shown that alloys or iron and zinc containing more than 22 per cent. Fe cannot be made without the imposition of pressure. For this reason Bablik not only doubts the theoretical possibility of Γ in normal hot dipped coatings, but suggests that the presence or absence of "pal" is governed by the pressure conditions obtaining in the alloy layers. He shows that an alloy containing 5·7 per cent. Fe when cast and having a specific gravity of 5·5 suffers a 20 per cent. volume increase (specific gravity 4·4) on being annealed for 3½ hours at 430 deg. Cent., thus indicating the probable sensitivity of these alloys to pressure conditions. Bablik suggests that the intermediate layer "pal" appears only when pressures are present, and that when compounds are free to grow unhindered as on convext surfaces, then "pal" disappears. This interesting theory requires much more attention, and may lead to profitable conclusions if applied to the iron-arsenic system and other similar systems.

Concerning the much-neglected iron end of the iron-zinc system, Svechnikov and Gridnev[11] agree with Jones[12] that the diagram must embrace a gamma loop. Schramm's diagram is constructed quite differently and includes an eutectoid. The Russian workers followed the technique elaborated for this type of problem by Jones and sintered mixed iron and zinc powders in iron capsules.

It will thus be seen that from the iron to the zinc end of this diagram confusion and contradiction still prevail. Apart from its theoretical interest, this work has serious practical importance in connection with the galvanising industry. Considerable caution must obviously be attendant upon any attempt to accept published figures concerning rates of attack until a more satisfactory state of affairs has been reached.

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Recent German Advances in Spectrographic Analysis.

By E. H. S. VAN SOMEREN, B.Sc.

A LARGE number of papers dealing with various aspects of spectrographic analysis have appeared in in the German technical Press in the last few months and a list of some of them is appended to this article. They are symptoms of a growing interest in the subject fostered by an admirable co-operation between

the development departments of apparatus manufacturers and the research staffs of technical institutions and metallurgical laboratories.

There has been no marked recent increase in the scope of spectrographic analysis. The method was first used for the sun, the alkaline earths, and the stars; then extended to the analysis of minerals, and later of metals. Soon it was applied to agricultural products and medical problems, such as the examination of animal tissues. By 1935 it had appeared in forensic laboratories and in detective fiction. An analytical method which has dealt with so much of the universe can only enlarge its scope slowly in future; its application to archæology and numismatics remains to be extended.

Of the papers under review, one by R. Ramb[1] refers to a selection of problems studied for various firms by way of preliminary inquiry of whether or not spectroscopy would deal with their special problems. The applications are largely metallurgical, and include the quantitative analysis of aluminium in a chromium-iron alloy, where it was found that 3–6 per cent. could be determined with a mean error of 4·2 per cent., and the estimation of traces of silicon in steel, where it appears likely that chemical tests are no more reliable than spectrographic ones, unless carried out with more than ordinary skill. An unknown complex alloy steel was qualitatively analysed, faults in the finish of a rolled zinc strip were identified as iron inclusions, the coating of a small sample of plated iron was found to contain zinc and cadmium, with traces of aluminium, copper, magnesium, manganese, nickel, and calcium, some of which may have been impurities in the iron itself.

Non-metallic samples examined include a group of specimens of cellulose, in which the metallic impurities were to be compared, some samples of rubber and of rubber fillers, of red lead as a raw material for glass manufacture, and a powdered mixture of rare earths which were all identified.

The range of samples dealt with in this paper is reminiscent of that met with by the writer some years ago in an average three months' work as spectroscopist to a large metallurgical firm. Numerous samples sent in by customers for matching had to be identified, often complicated by a request from the customer that the sample should be returned. That is where the spectroscopist scores over the chemist—for he can analyse a sixpence and spend it afterwards. Red lead figured as a substitute for cayenne pepper in some sandwiches which had been interfered with by a practical joker, and rare earths were encountered in the alloy which provides the sparks in a patent lighter. An unexpected metallic inclusion found in a bar of chocolate proved to be a dental alloy, not a piece of machinery.

Professor Gerlach, one of the pioneers of quantitative spectrographic analysis, and one of his collaborators review some recent advances in the technique of the method of which they have personal experience.[2] The attention given in this paper to the interrupted arc (*Abreissbogen*) does something to counterbalance the almost exclusive concentration on spark methods which is typical of recent German work. The interrupted arc is produced by a mechanical device which carries one electrode of the arc and moves it rapidly so as to make contact with the other electrode, and then draw back so far that the arc voltage is insufficient to span the gap and the arc goes out. Frequently the electrodes are dissimilar, only one forming the sample to be analysed, and the other being of some suitable metal, such as gold or copper or a purer specimen of the metal being tested. This method has the advantage that larger currents can be used with alloys of low melting point than is otherwise convenient.

It is claimed here that this technique produces spectra in which the banded background to the lines has a lower relative intensity, and consequently smaller traces of some impurities can be determined. The effect of changing the polarity of the arc, of changing the temperature of the electrodes (controlled by an auxiliary heating coil), and of running the arc in gases other than air, is discussed in detail. The interrupted arc is also applicable to non-metallic samples, such as salts on carbon electrodes, and full details for its use in this field are given, including two interesting examples. One is a museum specimen of ruby glass, from which a few particles were obtained by scratching a flaw with a diamond. These were analysed, and showed the specimen to be of glass whose colour was due to gold. The other sample was a metallic particle which had lodged in a workman's eye, which was found to be lead with antimony in it. The origin of the injury to the eye was questioned by an insurance company.

The interrupted arc technique has found no favour in England, partly because industrial spectroscopists here dislike extra complications of apparatus, partly because of the difficulties (not mentioned here) arising from its use. It adds two variables to the arc, that of frequency of interruption and that of ratio of duration of burning of arc to duration of total exposure time, and even without these, considerable patience is required to secure uniform spectra by the arc method. The reduction of relative intensity of the banded background to spectra can be partially attained (as Professor Gerlach points out here and the writer found in 1932) by the use of slow, contrasty photographic plates instead of the normal type of plate. As for sensitivity, in a fusible metal like zinc, the elements lead, cobalt, copper, nickel, and cadmium can all be detected in concentrations down to 0·0001 per cent. by the ordinary arc method.

The technique suggested here for running the carbon arc in carbon dioxide instead of air is recommended for the interrupted arc, but would, of course, also be applicable to the ordinary arc. The detection of the elements thallium, lead, iron, chromium, cobalt, and nickel in non-metallic samples is hindered in the carbon arc because of the bands due to air which mask the most sensitive lines of these elements; by enclosing the arc in a tube with a quartz window and an atmosphere of carbon dioxide, the detection of these elements is facilitated in smaller quantities; for example, in a salt containing iron, one twenty-thousandth of a milligramme of thallium was detected in this way.

Another point made here is that if the presence of a sensitive line (Tl 3776, for example) in a banded spectrum is doubtful to visual inspection, it may sometimes be verified by micro-photometric examination of the plate, either with a recording or non-recording microphotometer.

Professor Gerlach reports on a further refinement of the arc method suggested by K. Pfeilsticker,[3] which consists of an arc circuit with so much resistance in series with the arc that an arc cannot be maintained with the electrode gap normal (say, 3 mm.). An associated Tesla circuit provides a H.F. discharge across the arc gap which ionises the air space, so that the arc burns for a moment, then goes out, because a commutator in the arc circuit breaks off the arc voltage for, say, one-quarter second out of every

half second. This has all the advantages of the interrupted arc, except that the suppression of banded background is less complete. It has the additional advantage that it can be used to operate an arc where only A.C. is available, as the H.F. circuit will ionise the gap after each reversal of phase has extinguished the arc. Metals, such as aluminium, which are too easily oxidised to be arced otherwise, can be made to give arc spectra in this way. This new method, which might be called the ionised interrupted arc in English, seems to be a valuable advance in spectrographic technique, and it is to be hoped that the electrical complications will not hinder its development in this country.

Finally, this comprehensive paper discusses two methods of quantitative spectrographic analysis based on the widening of over-exposed spectrum lines instead of their blackening. This section will appeal only to readers familiar with densitometry, and outlines a valuable new technique for comparing the intensity of two spectrum lines which should be particularly useful for the analysis of the major components of a mixture.

Another paper, by H. Kaiser,[4] dealing particularly with light metal analysis, returns to the spark technique which is favoured by most German spectroscopists, and elucidates several points about the details of the spark discharge between metallic electrodes. First the author emphasises the value of the "Feussner" spark circuit. This circuit adds to the secondary spark circuit a commutator which regulates the number of H.F. oscillations which bridge the spark gap after each reversal of phase of the primary alternating current. This refinement, or complication, has proved its value in obtaining more uniform spectra, and its method of operation is made clearer by photographs of discharges. The effect of changing the shape of the electrodes is also discussed, and illustrated by photographs. This leads naturally to the question of "*abfunkeffekte*," or changes in the spark spectrum during the first few minutes of sparking. These effects, which are also dealt with in Gerlach's paper and in that by H. Winter,[5] are a very important source of error in quantitative spectrum analysis.

There is no English phrase for *abfunkeffekte*, for, although they have been observed by English workers, they have been dealt with empirically in the course of preliminary standardisation of spark conditions. By analogy we may call it "sparking fatigue." The typical symptoms of spark fatigue are only detected when successive exposures are made on a spark without interrupting it, and the relative intensity of a pair of lines is plotted against time. The curve will fall or rise steeply for from one to five minutes, then maintain an equilibrium value for some minutes, and possibly deviate again after a time too long to be of much importance to the routine spectroscopist. Sometimes the initial change is a sharp fall followed by a gradual rise before the steady value is obtained.

The important thing for the practical spectroscopist is that his standard conditions for sparking should be such that he is working on the flat part of his sparking fatigue curve for all the pairs of lines whose intensities he is comparing, and for all the samples of similar material; otherwise the accuracy attainable in his analysis is reduced. At this point it should be noted that German workers, notably H. Kaiser,[6] are beginning to specify accuracy, not by a rough percentage error based on comparison with chemical methods, but by the standard deviation of a group of repeated tests on the same sample, an application of statistical methods which is very valuable, because the probability that the error of a single result exceeds this standard deviation by various degrees is calculable.

In the paper by H. Winter[5] a series of sparking fatigue curves for a pair of aluminium and silicon lines in a series of aluminium-silicon alloys is shown, and the author produces a satisfactory theory connecting the variation in the shape of these curves with the variations in the crystal structure of the alloys. The effect of modifying the alloys with sodium and the effect of the constitution of magnesium-aluminium alloys on the sparking fatigue is also discussed. This very interesting paper also discusses the spectrographic analysis of the zinc die casting alloys by spark methods with a non-recording microphotometer for comparing selected lines of the spectra. Remarkably accurate results are claimed, whose errors are comparable with those of well-controlled routine chemical analysis.

An interesting series of photomicrographs shows the effects of sparking on silicon-aluminium electrodes, and the local modifications of micro-structure due to melting and supercooling of a "crater" of metal at each end of each spark are established. It is surprising that no reference is made here to the work of H. Triché,[7] who used the spark on chromium-aluminium alloys to detect local variations in composition, and also oxidised over the aluminium surface with a solution which left the chromium-rich constituent unchanged and carried out a quantitative spectrographic estimation of the chromium in the unoxidised crystals, which was constant even when the total chromium in the alloy varied from 2·4 to 4·7 per cent.

Since it has been claimed by critics that the accuracy of spectrographic analysis would be limited by the coarse micro-structure of certain alloys, it is important that the effect of micro-structure on the spark should be explored in this way, and the limits of the disturbance assessed. Detailed research of the kind summarised here is essential before the spectrographic method can displace chemical tests in routine industrial analysis instead of simply supplementing the work of the chemist, as is more usual at present. The close linking between spectroscopy and metallography in the paper by H. Winter makes it a noteworthy contribution to metallurgical analysis.

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Magnetic Properties of Nickel-Copper-Iron Alloys.

IN view of the remarkable magnetic properties of the nickel-iron alloys, and the recognised importance of precipitation hardening on magnetic properties, attention is constantly being given to the effect of the addition of a third metal which will induce precipitation hardening in the nickel-iron system. One of the most important of these is aluminium.[1] Another is copper, and the constitution of these alloys in

[1] THE METALLURGIST, 1935/36, 10, 39.

relation to composition and heat treatment has been dealt with in a previous article.[2]

The limited solid solubility of copper in iron is considerably increased by addition of nickel, and the difference between the solubility limits at high and low temperatures is slightly increased. Thus the maximum percentage of copper in solid solution at 1250 deg. Cent. is 11 per cent. with 0 per cent. of nickel and 22 per cent. with 25 per cent. of nickel,

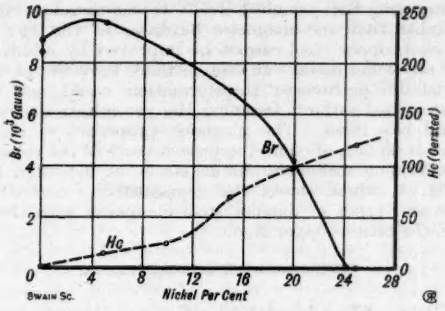

FIG. 1—*Influence of Nickel on the Remanence and Coercivity of Iron.*

while the corresponding values at 400 deg. Cent. are less than 1 and 8 per cent. of copper. In the plain copper-iron alloys hardening by quenching and reheating is brought about by precipitation of copper from solid solution only, no martensite being formed; on the other hand, the hardening of the nickel alloys by heat treatment is due to martensite or martensite-austenite formation, according to the nickel content. In the copper-nickel-iron alloys both processes, pre-

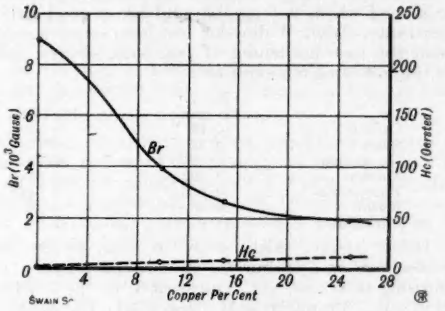

FIG. 2—*Influence of Copper on the Remanence and Coercivity of Iron.*

cipitation hardening and martensite hardening, can occur simultaneously and the effect on the magnetic properties, remanence and coercivity, has recently been shown in a paper on the "Magnetically Hard, Low-carbon Copper-Nickel Steels," by H. Legat,[3] to be much greater than a purely additive effect of these two processes. The material investigated contained less than 0·05 per cent. of carbon, with 0 to 25 per cent. of nickel, and of copper, and other additions in special series of alloys.

The character of the hardening curve is determined chiefly by the nickel content, which also determines the structure of the quenched alloys. The best composition is in the martensite-austenite range, the austenite fraction being large and capable of undergoing transformation. In the quenched state the remanence and coercivity are both low; on reheating, however, part of the austenite is converted into stable martensite and the remanence rises considerably, but the coercivity only slightly. At somewhat higher temperatures (around 700 deg. Cent.) the rate of internal diffusion rises rapidly and the

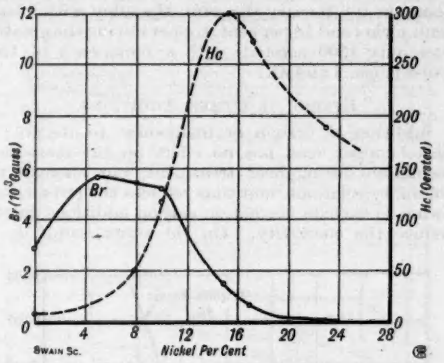

FIG. 3—*Influence of Nickel on the Remanence and Coercivity of Iron with 14 Per Cent. of Copper.*

copper-rich phase begins to precipitate, the resulting change in concentration inducing transformation of the remainder (*i.e.*, the greater part) of the austenite. Under the influence of these processes the coercivity rises steeply to a maximum and then decreases again, while the remanence falls to a minimum at maximum coercivity and rises again as the coercivity decreases. Since the temperature range in which these effects occur is very narrow, time and temperature of annealing are not interrelated as is usually the case in

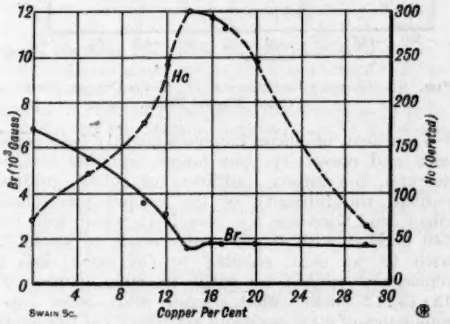

FIG. 4—*Influence of Copper on the Remanence and Coercivity of Iron with 15 Per Cent. of Nickel.*

precipitation hardening. The best quenching temperatures lie between 1100 deg. and 1300 deg. Cent., and the best quenching medium is cold water.

In the hardened state the coercivity of plain nickel steels rises almost linearly with the nickel content to 12 per cent., then more rapidly to 16 per cent.; with further increase in the nickel content the coercivity increases at about the same rate as for steels with less than 12 per cent. nickel. The reman-

[2] THE METALLURGIST, 1935/36, 10, 104.
[3] *Metallwirtschaft*, July 23rd, 1937, 16, 743.

ence rises to a feeble maximum at 4–5 per cent. nickel and then falls fairly regularly to almost zero at 24 per cent. nickel (Fig. 1). The coercivity of hardened copper steels rises linearly with increasing copper content, but does not exceed 10 oersted with 24 per cent. copper; the remanence falls sharply to 12 per cent. copper then more slowly (Fig. 2). In the copper-nickel steels in the hardened condition maximum coercivity is obtained with compositions lying on a line joining the 18 per cent. nickel, 0 per cent. copper point, to the 13·5 per cent. nickel, 25 per cent. copper point, in the ternary diagram, the alloy with 15 per cent. nickel and 14 per cent. copper having the greatest coercivity (300 oersted) with a remanence of 1500 gauss (Figs. 3 and 4).

Effect of Other Additions.

Addition of carbon or manganese to the 15 : 12 nickel-copper steel has no effect on the martensite formation during heat treatment, but changes the solubility relations, and thus reduces the remanence; owing to carbide formation carbon additions greatly reduce the coercivity. On the other hand, up to

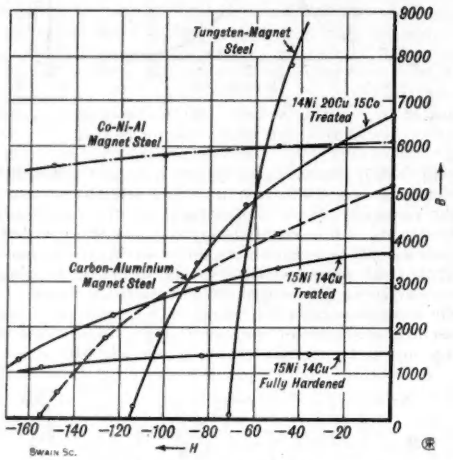

FIG. 5—*Demagnetising Curves of Nickel-Copper Steels and Other Magnet Steels.*

0·6 per cent. of silicon increases slightly both remanence and coercivity, but larger amounts seriously decrease the latter. Addition of cobalt gradually reduces the intensity of the copper precipitation effect, and therefore the coercivity which falls from 240 oersted with 0 per cent. cobalt to about 105 with 10 per cent. cobalt; at the same time the remanence rises from 2900 to 6000 gauss. The 15 : 15 : 12 nickel-cobalt-copper alloy steel has a remanence of 9000 gauss and a coercivity of 103 oersted. Addition of silver to the 15 : 12 : 3 nickel-copper-cobalt steel increases the hardening effect due to copper precipitation, and therefore increases the coercivity, but decreases the remanence, so that the product $B_r \times H_c$ remains practically constant. A maximum of 2·5 per cent. silver can be retained in solid solution at quenching temperature, but practically all is precipitated with the copper on reheating.

Two precipitation hardening effects can be obtained in nickel-cobalt-copper shells containing about 9 per cent. of molybdenum, one due to separation of the copper-rich phase, the intensity of which is increased by the presence of molybdenum, and the other, at higher temperature, due to separation of the molybdenum-nickel-cobalt phase. Since the maximum effects of these two processes do not occur simultaneously, no appreciable improvement in the magnetic hardness is obtained. Replacement of the molybdenum by tungsten, vanadium, titanium, or zirconium leads to no better results, since all these elements restrict the extent of the γ range, and decrease the stability of the martensite produced by tempering the quenched steel. It was therefore concluded that the magnetic hardness of the 15 : 15 nickel-copper steel cannot be improved by addition of any other metal. In each instance the effect of the austenite-martensite transformation could not be intensified without lessening the precipitation effect, and *vice versâ*. The magnetic properties of these steels do not, of course, approach those of the cobalt-containing nickel-aluminium steels, as indicated by Fig. 5, which shows the demagnetising curves of several types of magnet steel compared with those of the nickel-copper steels.

Case Hardening of 14 per cent. Chromium Steels.

To withstand wear, especially at raised temperature, it has been customary to make use of high-chromium high-carbon alloys often containing 25 per cent. or more of chromium and 2 to 3 per cent. of carbon. These steels are not forgeable and must be used in the cast condition. They possess a remarkable resistance to wear by attrition, but on account of their brittleness they are unsuitable for use under conditions involving impact stresses of any magnitude.

In order to secure similar wear-resisting properties in a steel which is forgeable and has a good impact resistance, Erich Widawski[1] has been experimenting with the case hardening of two basic electric steels of the following composition :—

	No. 1. Per cent.	No. 2. Per cent.
Carbon	0·12	0·45
Silicon	0·32	0·57
Manganese	0·42	0·72
Sulphur	0·023	0·028
Phosphorus	0·035	0·017
Nickel	0·53	0·37
Chromium	13·9	13·2

Discs, 10 mm. thick, were cut from 40 mm. diameter bar and carburised in two proprietary carburising mixtures, viz., Durferrit I, of the Durferrit G.m.b.H., Frankfurt a. M., and CMD 13, made by C. F. Goerig, Mannheim, and in a 50 : 50 mixture of wood charcoal and barium carbonate with the addition of 1 per cent. of soda. The carbon content of the case and the depth of penetration of carbon were studied by the examination of successive layers. By the use of a carburising temperature of 1000 deg. Cent. or above, the carbon content of the surface reached a maximum value of 3 per cent. The depth of penetration of carbon was about the same under similar conditions in both steels. After carburising at 900 deg. Cent. the total depth of penetration was about 1·2 mm., and was the same whether the time of exposure was ten or twenty hours. The total depth was not altered if the ten-hour period was succeeded by six hours in a fresh sample of the

[1] *Archiv für das Eisenhüttenwesen*, October, 1937, 1937–38, 11, 195.

carburising mixture, though the outer zone of higher carbon content was slightly increased at the expense of the inner zone of the carburised layer. After carburising at 940 deg. Cent. there was not much change in behaviour; the depth of case was 1·5 mm. and increased time of carburisation had no appreciable effect on the depth of penetration or on the carbon content of the surface layer. Below 950 deg. Cent. the carburising action soon came to a stop. Carburising at 970 deg. Cent. for ten hours gave a depth of penetration of 2·3 mm.; at 1000 deg. Cent. for ten hours the following results were obtained :—

Carburising medium.	Depth of penetration, mm.	Max. carbon at surface, per cent.
C M D 13	3·0 to 3·2	1·1
Durferrit I	4·1	3·0
Charcoal and barium carbonate	4·0	3·0

Three successive ten-hour treatments in fresh supplies of Durferrit I made no difference to the maximum carbon content of the face, but increased the carbon at 1 mm. below the surface from 1·6 to 2·0 per cent., and it was estimated that the total depth of penetration was increased to 6 mm. or 7 mm. (no accurate deduction was possible on this point since the specimen, 10 mm. thick, was to some extent carburised throughout). Carburising in

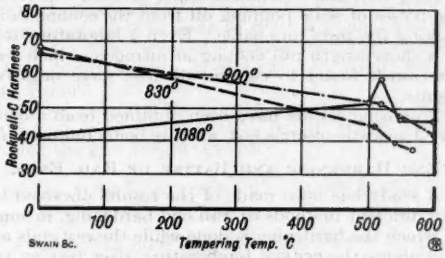

FIG. 1—*Surface Hardness of Steel* 1, *Carburised in Durferrit I for Ten Hours at* 1000 *Deg. Cent., Oil-hardened from* 830, 900, *or* 1080 *Deg. Cent., and Tempered at Different Temperatures.*

Durferrit I at 1100 deg. Cent. for 2½ hours gave a maximum carbon of 3 per cent. at the face and a total depth of penetration of 3·6 mm. to 3·8 mm. This high temperature, however, leads to the formation of a coarse carbide network, probably as a result of the precipitation of chromium carbide in the grain boundaries after saturation of the γ solid solution with carbon. To avoid undue grain growth a carburising temperature of 1000 deg. Cent. is probably most suitable.

Although the carburising medium employed in further work was Durferrit I, it may be noted that the charcoal-barium carbonate mixture gave similar results as judged by microscopical examination and appearance of fracture. These tests showed three zones in the carburised layer, and by the aid of microscopical observations interpreted in the light of the iron-carbon-chromium diagram of Tofante, Küttner, and Büttinghaus,[2] they were identified in the furnace-cooled material as follows :—

Zone 1 (*Outer Zone*).—Finely divided carbides $(Cr, Fe)_7C_3$ and $(Fe, Cr)_3C$ in a ground mass of chromiferous α iron.

[2] See THE METALLURGIST, August 28th, 1936, pp. 152–155. Fig. 3 on page 153 represents the constitution of the 15 per cent. chromium steels.

Zone 2.—A network of carbide $(Cr, Fe)_7C_3$, precipitated on cooling from the γ solid solution; extending in veins to—

Zone 3.—Chromiferous α iron and carbide, mainly $(Fe, Cr)_4C$.

The approximate carbon limits of these structures would be: Zone 1, above 1·6 per cent.; zone 2, from 1·6 to 0·8 per cent.; zone 3, from about 0·8 per cent. down to the carbon content of the original steel. The depth of the three zones was similar after carburising in the proprietary material, Durferrit I, and in the charcoal-barium carbonate mixture

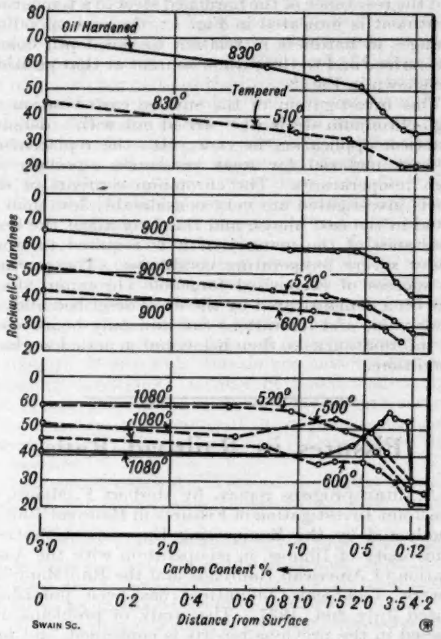

FIG. 2—*Hardness Throughout the Carburised Layer of Steel* 1, *Carburised in Durferrit I for Ten Hours at* 1000 *Deg. Cent., Oil-hardened from* 830, 900, *or* 1080 *Deg. Cent., and Tempered at Different Temperatures Between* 500 *and* 600 *Deg. Cent.* (*Full Line, Oil-hardened*; *Broken Line, Oil-hardened and Tempered.*)

(Table) and conclusions drawn from experiments with Durferrit I are therefore generally applicable to material carburised with the charcoal-barium carbonate mixture.

TABLE.—*Depth in Millimetres of Carburised Zones in Steel No.* 1, *Carburised at* 1000 *Deg. Cent. for Ten Hours.*

Carburising medium.	Durferrit I.		C–$BaCO_3$ mixture.	
	Steel No. 1.	Steel No. 2.	Steel No. 1.	Steel No. 2.
Depth of zone 1 ...	0·5	0·8	0·7	0·5
Depth of zone 2 ...	0·7	0·8	0·4	0·3
Depth of zone 3 ...	2·9	2·5	2·9	3·2
Total depth ...	4·1	4·1	4·0	4·0

The further work consisted of determinations of the effect of heat treatment on the case produced on the 0·12 per cent. carbon steel (No. 1) by carburising

in Durferrit I for ten hours at 1000 deg. Cent. The highest surface hardness[3] (68 Rockwell C, or 1050 D.P.H.) was obtained in the fully carburised steel by quenching at 830 deg. Cent. When higher quenching temperatures were employed the hardness was lower on account of the retention of austenite. On tempering, however, the steel hardened from 830 deg. showed a considerable and progressive fall in hardness; that hardened from 900 deg., a more moderate fall, and that hardened from 1080 deg. a rise from 42 Rockwell C (420 D.P.H.) to 59 Rockwell C (742 D.P.H.) on tempering at 520 deg. Cent. The effect of hardening temperature on the surface hardness and the resistance of the hardened steel to a tempering treatment is indicated in Fig. 1; the corresponding changes in hardness in relation to the depth below the surface and to the carbon content at that position are shown in Fig. 2.

This investigation of the surface carburisation of high-chromium steels was carried out with a definite practical application in view, viz., the replacement of cast material for wear resistance especially at high temperatures. The chromium contents of the steels investigated are very considerably less than is usual in the cast alloys, and this may affect the heat resistance of the material if it is required to work under severe temperature conditions. The promise of success of carburised forgeable chromium steels has been demonstrated by the work described above; their value and limitations can now only be assessed by information as to their behaviour in actual working conditions.

Fissures in Railroad Rails.

A THIRD progress report, by Herbert F. Moore, of the Joint Investigation of Fissures in Railroad Rails,* conducted by the Engineering Experiment Station, University of Illinois, in co-operation with the Association of American Railroads and the Rail Manufacturers' Technical Committee, has been published, dated July 2nd, 1937. The study of problems discussed in the previous reports is continued, and preliminary results given of tests of batter of rails at rail joints, of the effect of end hardening on batter, and of the strength and ductility of rail steel. The various problems are dealt with under the following headings.

FIELD TESTS FOR WHEEL LOADS IN SERVICE.

Results on further test rails (110 lb.) laid on a line carrying very heavy freight traffic in both directions are compared in a graph with results at the three other locations given in the previous reports.

The frequency of wheel loads of 40,000 lb. and over (which laboratory experiments had indicated as that liable to spread a shatter crack into a transverse fissure) was about 0·1 per cent. in two of the test locations and about 0·013 and 0·0017 per cent. respectively in the other two locations. In the latter two locations, wheel loads of 30,000 lb. and over showed approximately the same frequency (0·1 per cent.) as loads of 40,000 lb. in the two former locations, the average nominal loads being approximately in the same proportion. One wheel load of 65,000 lb. and another of 84,000 lb. were recorded during the latest tests, both at the same very stiff spot on the road bed, and at train speeds of 35 and 45 m.p.h. respectively. It is suggested that such high loads are the combined result of out-of-round wheels or flat spots coinciding with a hard spot in the track.

Comparative curves are also given showing the ratio of wheel load to bending moment on regular track and on bridges (the bending moment being an approximate measure of track stiffness), and it is remarked that while the direct action of wheel loads starts fissures, bending stresses play an important part in fissure growth, and therefore both factors must be considered in the likelihood of fissure development.

ACCEPTANCE TESTS OF RAILS.

Preference is again given for the slow, head down-bend test over the standard drop test as a detector of shatter cracks, and a preliminary sketch is given of a suitable bend test machine.

Further experiments with the microphone test as a non-destructive detector of shatter cracks during cooling were not successful, other factors, especially the noises of scale popping off from the cooling rails, making the tests unreliable. Even a laboratory test of a short length rail cooling in nitrogen, which was afterwards found to contain fissures, gave negative results.

Promising results have been obtained from a combined acoustic-electric test, and are being followed up

END HARDENING AND BATTER OF RAIL ENDS.

A study has been made of the results obtained by five different methods of rail end hardening, in some of which the hardening is done while the rail ends are still above the critical temperature after leaving the mill, and in others in which the rail end is reheated either electrically or by gas or oil burners. In some processes compressed air and in others water sprays are used as the quenching medium.

The hardened ends have been investigated by macro and micro examination, by hardness exploration by the Rockwell C method over a longitudinal section, and by tensile, fatigue, and impact tests of the hardened end and normal rail metal. The length of the hardened portion on the tread averaged about 4in., and the hardness in the hardened zones ranged from 31 to 49 Rockwell C, as compared with 23 to 31 Rockwell C in the unhardened rail heads. A chart is shown giving an experimentally obtained curve for the conversion of Rockwell C values to Brinell for rail steel, and a mean factor of 9 deduced from the chart has been used for subsequent conversions.

The reproduction of the micrographs of the treated rails in the report is unfortunately too poor for any comment to be made on the structures obtained, but it is stated that in one or two cases examined there was evidence of localised sections in which the structure was martensitic.

The comparative physical tests from the hardened and unhardened portions showed that for all rails so tested, the increase in tensile strength in the hardened portion was accompanied by increase of elongation and reduction of area and by greater toughness, as shown by impact tests. This is commented on as "a striking illustration of the possibility of increasing the strength of high-carbon steel without reduc-

[3] The following equivalents of Rockwell and Diamond Prism hardness given in the American Society for Metals Handbook, 1936, may be useful in connection with Figs. 1 and 2:—

Rockwell C...	20 ...	30 ...	40 ...	50 ...	60 ...	70
D.P.H.	... 223 ...	287 ...	389 ...	540 ...	765 ...	1150

* Published by the University of Illinois, Urbana, Illinois, U.S.A., pages 29, price 15 cents. The previous reports were reviewed in THE METALLURGIST, February 28th, 1936, page 107, and December 25th, 1936, page 179.

tion of ductility by proper heat treatment," but would appear to follow naturally with correct treatment, especially following a reheating of the rail end from below to above the critical temperature, which has a definite refining action on the coarse structure left from the finishing temperature at the rolls.

Batter tests have been made with the rolling load testing machine described in previous reports, two short lengths of rail being connected together by a joint bar. The results given, as tabulated below, are somewhat surprising in that the amount of batter appears to bear no relation to the hardness.

Process No.	Rail section.	Condition.	Hardness, Rockwell C.	Batter after 2,000,000 passages of a 40,000 wheel load.
5	112 lb. per yard	Hardened ...	45	0·004
	112 lb. per yard	Unhardened	30	0·009
2	131 lb. per yard	Hardened ...	32	0·003
	131 lb. per yard	Unhardened	24	0·0075
3	131 lb. per yard	Hardened ...	38	0·006
	131 lb. per yard	Unhardened	29	0·016

The hardness figure of 24 for the unhardened rail in process 2, given as equivalent to 216 Brinell, appears unusually low for a rail of standard American composition, but in spite of this gave less than half the batter than the rail of the same section in procees 3, whose hardness was 29 Rockwell C, equivalent to 258 Brinell.

Curves given of the growth of batter with cycles of wheel load indicate that batter occurs chiefly during the first 500,000 cycles.

Temperature Limits for Controlled Cooling of Rails.

A study has been started of the correct temperature limits for the controlled cooling of rails necessary to eliminate shatter cracks, and also of the amount by which the normal rate of hot bed cooling must be diminished. Rail lengths of 1ft. to 2ft. will be cut off at the hot saw and placed in boxes of varying heat insulation. The initial temperature of placing in the boxes and the final temperature of removal will be varied in different series. The results obtained from these tests should be of very great interest, although the present temperature limits employed in American rail mills as specified in the Sandberg patents have proved a definite prevention of shatter.

Production of Shatter Cracks by Treatment with Hydrogen.

In view of work in other countries tending to show that "flakes" or shatter cracks can be produced in various steels by heating in hydrogen, a series of experiments was carried out in which 6in. lengths of rail steel known to be free from shatter were heated in hydrogen for 27-hour periods at temperatures from 2100 deg. Fah. (1150 deg. Cent.) to one sufficient to melt the surface. The samples were subsequently cooled in still air, by a fan, or by water quenching from 820 deg. Cent., and in all cases shatter cracks were found after subsequent sectioning and etching. It is considered that these experiments offer reasonable proof that hydrogen can be one factor which produces shatter cracks or flakes in steel, but do not justify the view that hydrogen is the only factor. The explanation of the mechanism of the formation of cracks due to occluded hydrogen, based on the lowering of its solution pressure and diffusibility in steel with falling temperature and the consequent setting up of heavy internal pressures, is considered logical, but it is emphasised that whether hydrogen pressures or localised thermal stresses be the cause of shatter, controlled cooling should act as a preventive.

It appears to the reviewer that another explanation of the effect of occluded hydrogen may be in embrittling or weakening the structure—especially around the crystal boundaries—thus enabling the thermal stresses to produce ruptures more readily.

Miscellaneous Special Tests.

Under this heading brief accounts are given (*a*) of the failure by transverse fissure of four rails made of Bessemer steel, two rolled in 1909, and the other two of unknown age; (*b*) of the investigation of two rails removed from track owing to a "cold shut" or longitudinal seam at the top of the web; (*c*) web failures in rails within the joint bars chiefly due to segregation; (*d*) service failures of special test rails laid in track, all of which were from heats for which etch tests had shown pronounced shatter cracking; and (*e*) "damage line" stresses of rail steel at low temperatures, *i.e.*, results obtained by fatigue tests run above the endurance limit for a number of cycles, and then at the endurance limit for a subsequent number of cycles. Results are given showing the "damage resistance" for various rail steels, both shattered and shatter free, at temperatures from 70 deg. to 40 deg. Fah., but do not indicate any outstanding variations. It may be noted that in this list of tests none of the controlled cooled rails showed shatter cracks, while some of the hot bed cooled rails showed many shatter cracks and the normalised rails a very few.

The report concludes with an appendix giving a formula for determining the shearing stress in the web of a rail.

Considering the importance of the work with which these reports deal, it seems desirable that more detailed and fuller accounts should be given of many of the experiments which have been carried out.

This and previous reports give the impression that controlled cooling as a positive prevention of the formation of shatter cracks in rails is still in the experimental stages, whereas all American rail mills are now equipped to carry out the process, and about 700,000 tons of rails have been treated. Some of these have been in track for over four years without failures from transverse fissures being reported.

Cerium.

Attention has again been drawn to possible extensions in the use of cerium in an article on "Cerite Metals," by R. Strauss.[1] The term "cerite metals" refers to the mixture, sometimes known as "Mischmetall," of cerium with other rare earth elements usually containing about 50 per cent. of cerium, 40 per cent. of other metals of the cerium group (lanthanum, 25 per cent., samarium, neodymium, and praseodymium), and the remainder yttrium elements and iron. The mixture is obtained by electrolysis of the molten chlorides of the cerium

[1] *Metallwirtschaft*, September 24th, 1937, 16, 973.

earths from monazite sand, which is valuable mainly for the thorium that it contains. The product obtained from the cerite residues naturally varies somewhat in composition, containing from 40 to 60 (but rarely more than 60) per cent. of cerium.

By far the most important application of cerium is in the manufacture of pyrophoric alloys for pocket lighters and other ignition devices. Mischmetall readily emits bright, very hot sparks when struck or scratched by steel. The cerite metals are, however, too soft for technical use, and, long ago, Auer von Welsbach proposed the use of cerium-iron alloys containing 15 to 40 per cent. of iron for this purpose. An alloy with 30 per cent. of iron appears to be the most suitable. It may be made by the addition of iron, in the form of powder or fine turnings, to the electrolytic cell, or by adding iron to cerite metal which has been melted in a graphite crucible under a layer of sodium chloride, or preferably barium chloride, flux.

Many variations of the pyrophoric alloys have been proposed and employed. Incorporation of a proportion of cerium sub-oxide renders the alloy more resistant to atmospheric oxidation and improves its pyrophoric power. Alloying with boron increases hardness and tends to economy in consumption. Alloys with silicon have similar advantages, and that containing 8 to 10 per cent. of silicon and 5 to 7 per cent. of calcium is said to combine excellent pyrophoric properties with a high resistance to atmospheric influences. For pyrotechnic purposes the alloys with mercury are notable for their intensely brilliant display of light. Amalgams with 10 per cent. of cerium fire spontaneously in air, while the alloy with 80 per cent. of cerium gives the maximum brilliance of sparks.

The chief metallurgical interest in this review of the production, properties, and uses of the cerite metals, lies in the statements made with reference to the effects of cerium in the improvement of aluminium alloys and in the refining of steel. In the early days, relatively large additions of cerium to aluminium alloys were tried without much promise of success. H. W. Gillett and V. H. Schnee[2] expressed the view that cerium in aluminium alloys is "an expense not attended by any benefit." Meissener claimed that cerium decomposed the compound $FeAl_2$ and so improved the ductility of aluminium alloys containing iron. More recently the advantages of cerium in amounts up to 0·2 per cent. have been recognised, but in reference to these no mention is made of the Ceralumin series of alloys developed by Messrs. J. Stone and Co., Deptford. Details of the composition and properties of these alloys are readily available.[3] The cerium (introduced as mischmetall) is said to have a refining influence on the micro-structure, thus behaving in a similar manner to titanium, and to permit the beneficial effect of high iron content (1·0 to 1·4 per cent.) to be obtained in the quenched and aged alloy, whether cast or forged, by suppressing the formation of the brittle iron-aluminium constituent.

In connection with the influence of cerium in light alloys, no mention is made of the promise shown by certain cerium-magnesium alloys described by Haughton and Prytherch,[4] in which the mechanical properties at elevated temperature are improved by the addition of small amounts of cerium.

[2] *Journ. Industrial and Eng. Chem.*, 1923, 15, 709.
[3] *E.g.*, in "The Metal Industry Handbook," 1937, pp. 192–193.
[4] "Magnesium and Its Alloys," H.M. Stationery Office, 1937, p. 55, *et seq.*

In discussing the effect of cerium on steel Strauss refers optimistically to its use for removing, or reducing, the amount of oxygen, nitrogen, sulphur, and phosphorus. He states that the I.G. Farbenindustrie have demonstrated the advantage of using not cerite metals alone, but an alloy containing 5 to 15 per cent. of cerite metals, 25 to 60 per cent. of aluminium, 5 to 15 per cent. of calcium, magnesium, or silicon, 5 to 30 per cent. of titanium, remainder iron. This is introduced into the metal as powder or lumps. It would, however, be impossible to attribute to one of the constituents, *e.g.*, cerium, any effect which might be produced by the addition of such a complicated mixture of chemically active elements to steel. In the production of vanadium steel the use of an alloy of 20 to 40 per cent. of vanadium and up to 40 per cent. of cerite metals is advised, the vanadium thus being relieved of its scavenging action and left free to alloy with the steel.

In this section of the paper the considerable amount of work which has been done in attempting to determine the value of cerium as an alloying element in steel is not adequately reviewed. Additions of cerite metals to steel, though made with all possible care, show a high loss of cerium. When more than 0·25 per cent. of cerium has been added marked segregation has been observed; for example, a 70 lb. steel ingot contained 0·6 per cent. at the top and 0·3 per cent. at the bottom. Little has been added to our knowledge of the subject to justify any modification of the opinion expressed by H. W. Gillett and E. L. Mack in their book on "Molybdenum, Cerium, and Related Alloy Steels" in 1925. Tests on cerium steels are generally vitiated, as far as showing the alloying action of cerium is concerned, by the multitude of non-metallic inclusions present. Its successful use in eliminating impurities is dependent on finding a means of getting rid of the accompanying inclusions. Gillett and Mack's statement still holds that "cerium appears to have no true alloying effect on steel and to do no good." Metallurgical application of cerium in the steel industry seems to be as remote as ever.

Books and Publications.

Effect of Impurities in Copper. By S. L. ARCHBUTT, F.I.C., and W. E. PRYTHERCH, M.Sc. (with a foreword by Dr. H. W. BROWNSDON and an introduction by Dr. C. H. DESCH, F.R.S.). Research Monograph No. 4. Pp. xvi+134, with 44 illustrations and 51 tables. London: British Non-Ferrous Metals Research Association. 1937. 12s. 6d. (postage 6d.).

COPPER, one of the earliest known metals, with a history dating back to Neolithic times, is still unsurpassed in its beauty and scope of utility, and is, indeed, probably the most essential metal in these days of electrical development. The literature on copper is enormous and practice in extraction and manufacture over some 6000 years has accumulated a great deal of knowledge, but it has remained for the present century to begin the systematic investigation of the common impurities.

This, the fourth of a series of books in which the B.N.F.M.R.A. has published condensed accounts of its major investigations, is a worthy addition to the

publications dealing with copper. It represents mainly a digest of a number of reports, several of which have been published at intervals by the Institute of Metals, describing a survey of the effects of impurities associated with commercial varieties of copper. These reports have been suitably grouped and additions made of connected information from other sources. The work and its implications can thus be viewed conveniently as a whole, and there is no doubt that its value in this form is much greater than could be drawn from the various scattered sections. The investigation, commenced some years ago under the direction of the late Dr. W. Rosenhain and only recently terminated, has accumulated the contributions of a number of scientists, of which Mr. Archbutt has been a continuous member, and he is thus the natural author of the collected results. The programme entailed the study of the effects produced by various single elements, starting with oxygen, iron, bismuth, arsenic, antimony, nickel, lead and phosphorus, and then certain of these in pairs, followed by oxygen together with bismuth and arsenic, bismuth and antimony, nickel and antimony, and nickel and arsenic. Finally a set of three and one of six impurities together with oxygen were taken. The methods of examination included the study of rolling properties, tensile, hardness, impact resistance and fatigue tests, and determinations of density and electrical conductivity. In most cases the constitutional aspects of the various impurities are discussed and useful general statements are given regarding, for example, the beneficial effects of other elements in the presence of bismuth, the undesirability of nickel, together with antimony and oxygen, and the unrecognised virtues of antimony. Chapters are added dealing with works experiments and segregation but this part of the work was not carried very far.

It will be clear to the reader that the task of remodelling separate rigid reports into the rounded form of a book in uniform style has been one of compromise and some difficulty, and the authors are to be congratulated on their presentation of such a consolidated view of their work. Chapter I, however, begins baldly in report style, and some twenty pages are passed before the text begins to flow easily. In this respect the book improves as it progresses, ending with a most concise and interesting chapter on the comparative effects of impurities on mechanical and physical properties of copper.

The impression is gained that the book is addressed to those already fairly well acquainted with the metallurgy of copper, but the opportunity might well have been taken to make it interesting to a less limited field of readers and of greater appeal to those needing direct advice on practical questions concerned in both manufacture and use. One might look, for instance, for an introductory chapter, including the matter given in Chapter II (Commercial Varieties of Copper), with a discussion of the practical view of the common impurities and the development of the plan of research. An expansion of the concluding chapter, linking up the results of the research with practical metallurgical considerations and with the properties required in copper for different types of use would also have wide possibilities and a welcome from the reader seeking assistance in dealing with the drawing up or meeting of specifications.

Such criticisms do not affect the value of the matter presented. Within the plan the abridgment has been nicely controlled, and it is only in the important sections describing the melting and casting methods that the reader may possibly wish for more explanation. The authors have given a large amount of detail, represented by fifty-one tables with adequate illustrations, which make the book valuable for reference purposes. As a last, but important, feature the book is splendidly arranged and printed and has obviously been edited and proof-read with the complete efficiency which is to be expected in the B.N.F.M.R.A. publications. The book should be of considerable value to all interested in copper, particularly those concerned in its production and manufacture.

Steels for the User. By R. T. Rolfe. Demy 8vo, pp. ix+280. London: Chapman and Hall, Ltd. 1937. 21s.

The engineer or user of metals frequently finds that the standard metallurgical textbooks are unsuited to his immediate need for a book of reference which will provide adequate information about the properties of the materials in which he is interested, combined with a sufficient explanation of the guiding principles of the subject to enable him to interpret and foresee differences induced by variations in composition, structure, treatment, &c.

In furnishing ample data of the type required, frequently based on examples from actual service, and always presented with a background of scientific reasoning, this book goes far towards meeting the need of the user in the field of the plain carbon steels. Mr. Rolfe has excellent qualifications for his task, as he is well known as a scientific metallurgist and is also a user in the sense that he has to exercise metallurgical control over the production of a large engineering works. Without going into detail, it may be said that the book is concerned on the one hand with carbon steel products, their composition and treatment, and on the other with methods of assessing their quality. Under the first heading special mention should be made of the comprehensive accounts of bright and free-cutting steels, of steel castings, and of the case-hardening process, which supplement the full general discussion of carbon steels. Under the second, one notable feature is the thorough consideration of the general principles of selection of materials based on previous discussion of methods of testing; and another is the extensive use of the microscope which is made throughout. In some cases (*e.g.*, crankshafts) the hope is expressed that micrographs will be used as acceptance tests and not taken merely for information.

The book is illustrated by some excellent photomicrographs taken in the author's laboratory by Mr. J. R. Bryant, and admirably reproduced by the publishers. Structural features are well correlated with properties. Constitutional interpretation of the micrographs is not always so consistently clear. Martensite may not "conveniently be regarded as a solid solution of carbon in β iron" (page 99), though it is only fair to say that the next clause throws some doubt on this conception, and the statement on page 139 completely rejects it. The further discussion of the lattice structure of martensite (page 112) is somewhat remote from the practical objective of the book. Then again, although a clear distinction is generally drawn between the results of cooling through, and of tempering below, the critical range, this distinction is not made quite clear in the case of the different varieties of "sorbite" (page 141). The point to be emphasised surely is that a lamellar structure, how-

ever fine, must always have been formed during cooling, and can never originate in a tempering treatment. Similarly massive or "free" ferrite is a product formed only during cooling, and in certain circumstances may remain incompletely dissolved during reheating before a further cooling (normalising or hardening). The ferrite of Fig. 27 has this appearance, though the description on page 100 is ambiguous as to its origin. These, however, are minor points in which descriptions otherwise clear and accurate may prove to be a little misleading to the general reader; but they illustrate the fact that expert interpretation will still be required even when micrographs attain to the status of acceptance tests.

As already mentioned, the present work deals mainly with carbon steels used in industry; alloy steels receive very little mention, except for specialised uses (*e.g.*, stainless steels, nitriding steels, &c.). To deal with a selected number of standard alloy steels on similar lines would require another volume, and it may be hoped that some day such a volume will be forthcoming.

Protective Films on Metals. By E. S. HEDGES. Second edition. Demy 8vo, pp. xv+397. 1937. London: Chapman and Hall, Ltd. Price 21s.

SINCE the first appearance of this book five years ago, much important information has accumulated on coatings applied for decoration or protection, and this part of the original edition was badly in need of revision. The book may be broadly divided into two parts, the first section, comprising six chapters, dealing with the mechanism of corrosion, with protective films formed during corrosion and with anodic passivity, has been brought up to date by the addition of notes and references on recent developments, but it is comparatively unchanged, the original length having been increased by only about 10 per cent.; the other section, on protective coatings, occupies the final seven chapters, and has been more than doubled in total length. One omission from the general scheme which might with advantage have been repaired in this second edition is an account of inhibitors used in pickling.

An author attempting a revision of a book is faced with a difficult task, since, with the accumulation of new facts of far-reaching importance, the viewpoint on the subject shifts, and processes appear in a new perspective. The choice is then between rewriting and merely adding to the existing framework, thereby endangering the balance or continuity of the book.

Certain sections of this second edition have been entirely rewritten and are admirable. This is especially true of the account of hot-dipped tin coatings which provides a clear picture of the processes of hot tinning (particularly of tin-plate manufacture) and of modern views on the structure and testing of tin coatings and on the corrosion of tin-plate with special reference to canning. The chapters on sprayed metal coatings and on paints, lacquers, and enamels have been considerably extended and largely rewritten, and they give a clear and useful general account of these subjects, special attention being given to lacquers for tin-plate and to vitreous enamels.

The chapter on electro-plating has been amplified chiefly by the inclusion of descriptions of the jet and chord tests for thickness determination of coatings and by the addition of notes on developments in the knowledge of the structure and adhesion of electro-deposited metals. Although most of the important developments which have occurred during the period under review are mentioned, the revised chapter gives a less clear and balanced picture of the present position than could have been made with the material available.

A more extended treatment has been given to methods of protecting aluminium and magnesium by anodic oxidation and by chemical treatments, and sections have been added on the protection of zinc and tin.

The book is clearly written and contains numerous references to original work. It provides a useful and up-to-date survey of the complicated subjects of corrosion and protective coatings, and it deserves a place on the bookshelf of the metallurgist and the engineer.

La Fatigue des Métaux. By R. CAZAUD and L. PERSOZ. 16 cm. by 25 cm. Pp. viii+190. Paris: Dunod. 1937. 75f.

THIS work of 190 pages contains much more than a general review of the subject of fatigue in metals. The authors have carried out a great deal of original research in connection with alloyed steels used in aircraft engines, and the results of their fatigue tests are given in tables that add much to the immediate practical value of the work. Results obtained by other investigators also are summarised concisely in tables and diagrams, and careful consideration is given to the work of English and other investigators. The volume contains a great deal of serviceable data.

The authors have succeeded also in presenting an admirable and very readable general review of the historical development and current knowledge of the subject, and the wealth of data contained in the tables and diagrams serves primarily to illustrate the lessons in this review. The work can be recommended to undergraduates and others who desire to approach the subject or advance logically on well-considered lines. Eight concise chapters deal with history and generalities, the character of fatigue fracture and its "mechanism" within the structure of the metal, methods and results of fatigue testing, the influence of diverse factors, such as corrosion fatigue, and with a variety of practical applications illustrating the practical importance of fillets, &c. Each chapter is followed by a brief bibliography that appears to be selected to facilitate further reading, and the volume is well illustrated with original photographs, graphs, and diagrams.

Joint Committee on Materials and their Testing.

IN the article on "Notched Bar Testing" in the October issue, the above Committee was described as an outcome of the London Congress of the International Association for Testing Materials. Actually, as reported in THE ENGINEER, April 23rd, 1937, page 465, the Joint Committee was formed before the Congress, it held its first meeting on January 12th, 1937, and at the conclusion of the Congress it was agreed that the existing Joint Committee should take over all matters concerning the International Association in the country.